08

Short-period multilayer mirrors for high-resolution multilayer mirror/crystal monochromator

© V.N. Polkovnikov,[1] N.I. Chkhalo,[1] R.A. Shaposhnikov,[1] A.D. Nikolenko[2]

[1]Institute of Physics of Microstructures, Russian Academy of Sciences,
607680 Nizhny Novgorod, Russia
[2]Budker Institute of Nuclear Physics, Siberian Branch, Russian Academy of Sciences,
630090 Novosibirsk, Russia
e-mail: shaposhnikov-roma@mail.ru

Received April 24, 2023
Revised April 24, 2023
Accepted April 24, 2023

The paper presents the results of studies of W/B_4C multilayer structures with small periods for their use as the first mirror in a two-mirror soft X-ray monochromator at the „KOSMOS" station of the VEPP-4M synchrotron. It is shown that even if the periods of the mirror and the RbAP crystal coincide, when operating in a wide wavelength range, the Bragg angle must be adjusted due to the stronger refraction in the W/B4C multilayer mirror.

Keywords: multilayer X-ray mirrors, synchrotron applications, X-ray monochromators.

DOI: 10.61011/TP.2023.07.56631.102-23

Introduction

A two-crystal monochromator [1] has been used for many years to conduct studies in the soft X-ray range at the „KOSMOS" station in the „Siberian Synchrotron and Terahertz Radiation Center (CUC „SSTRC") of Budker Institute of Nuclear Physics, Siberian Branch, Russian Academy of Sciences". Si(111) crystals in Bragg geometry with incidence angles close to normal are used for conducting studies with high spectral resolution in a monochromator. The scheme of a two-crystal monochromator is shown in Fig. 1.

The minimum energy available to a monochromator with such crystals is 2000 eV and allows working in the vicinity of *K*-edges of elements such as phosphorus and sulfur. *K*-the edges of such technologically important elements as aluminum, silicon, magnesium, fluorine, oxygen do not fall into the working area of the station. The possibility of replacing the Si monochromator with organic crystals with long periods is considered to expand the operating range into the long-wavelength region. Crystals of potassium biphthalate or rubidium (KAP and RbAP) are good candidates for this role. Thus, the KAP(001) crystal has a lattice period of 13.3 Å, which makes it possible to reach the working energy of the monochromator 470 eV and work on the K-edges of light elements up to oxygen. One of the promising tasks, in particular, is to study the properties of perovskites, which have oxygen in their composition with a hole on the *p* shell.

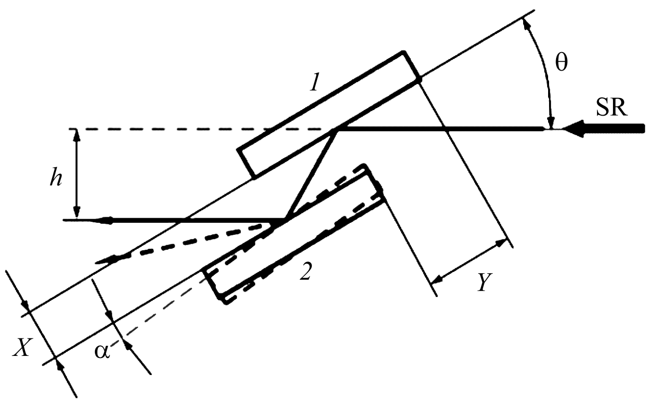

Figure 1. Diagram of a two-crystal monochromator used at the synchrotron radiation station „KOSMOS". SR — incident synchrotron radiation, *1* and *2* — monochromator crystals.

The problem of using biphthalates at synchrotron radiation stations is their low radiation resistance, which leads to degradation of the properties of the first crystal under a white synchrotron radiation beam (SR) in a few minutes. In this regard, it is proposed to use a hybrid optical scheme in which the first crystal is replaced by a multilayer mirror, implemented, for example, in [2,3]. Being the first element, the multilayer X-ray mirror (MXRM) reduces the intensity of the radiation incident on the crystal by many orders of magnitude due to the preliminary monochromatization of radiation at the level of $\lambda/\Delta\lambda \sim 200$, thereby increasing its lifetime. The spectral selectivity will be determined by the crystal at the output of the monochromator.

The specific feature of using multilayer mirrors for this task is the extremely small value of the period, at the level of 13 Å. The exponential nature of the impact of interlayer roughness on the reflectance requires atomically smooth surfaces. The required number of periods also increases to 300−500 due to the small size of the period, which limits the permissible instability of the technological parameters of the growth of multilayer mirrors. Also, a greater refraction in mirrors than in organic crystals should be expected due

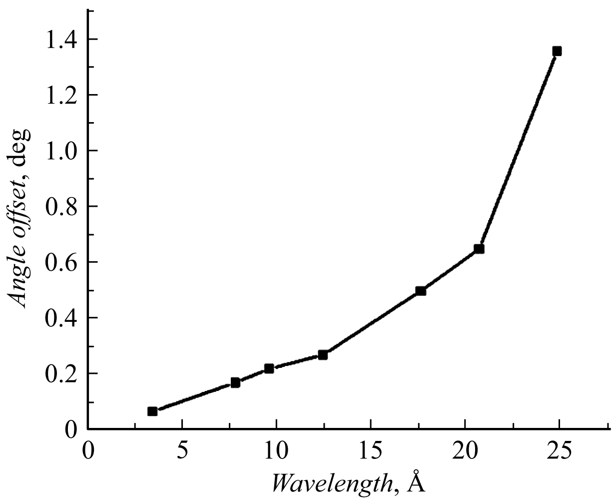

Figure 2. The dependence of the difference of the Bragg angles of the KAP crystal and the multilayer mirror W/B4C with the same periods on the wavelength. The proportion of layers W in the MXRM period $\gamma = 0.46$.

to the presence of „heavy“ material in the multilayer mirror period. The latter indicates the inevitable mismatch of the Bragg angles in the process of scanning for photon energies. All these factors are investigated for a short-period W/B4C mirror in this paper.

1. Selection of materials for a multilayer mirror

The wavelength range in which the optical scheme of the monochromator should be optimized for operation is 1.69–24.8 Å, which leads to the need for synthesis of short-period structures ($d \sim 1$ nm). It should be borne in mind that the maximum reflectance is determined not only by the optical characteristics of the mirror materials in the selected wavelength range, but also by the amount of roughness and transition layers, the development of which in the structure leads to degradation of the reflectance. It is also necessary to ensure the stability of the reflective characteristics of the structure used under the influence of thermal exposure. The study results show that the structures based on carbides [4] are the most advantageous from the point of view of thermal stability. Analysis of reflectances of short-period mirrors shows that multilayer mirrors based on tungsten and boron carbide [5–7] are promising structures for operation in this wavelength range. The results of the study of the effect of annealing on the reflective characteristics of W/B4C structures indicate their high thermal stabilitysti [8–10]. Based on this, it can be concluded that to replace the crystal in the optical scheme of the monochromator used at the synchrotron radiation station „KOSMOS“, a multilayer mirror based on tungsten and boron carbide should be used.

The values of the angles at which the maximum reflection is achieved at a given wavelength (first column), for KAP crystals (second column) and RbAp (third column)

Length waves, Å	Angle, degrees (KAP)	Angle, Degrees (RbAp)
24.80	68.80	
20.67	50.98	53.23
17.71	41.76	43.36
17.59	41.39	42.98
15.50	35.64	36.93
13.78	31.20	32.28
12.40	27.79	28.73
11.27	25.07	25.91
10.33	22.86	23.61
9.54	21.01	21.70
8.86	19.45	20.08
8.27	18.11	18.69
7.75	16.94	17.48
7.29	15.92	16.42
6.89	15.01	15.49
3.39	7.32	7.55
1.69	3.65	3.76

The table below shows the values of the angles at which the maximum reflectance is achieved for KAP crystals with a period of $d = 13.3$ Å and RbAp with a period of $d = 12.9$ Å.

It is worth noting that when replacing a crystal with a multilayer mirror, the problem arises of choosing the optimal mirror period, since the use of a structure with an identical period will lead to a displacement of the angle corresponding to the maximum reflectance at a given wavelength. This mismatch of angles for the KAP crystal and the W/B4C multilayer mirror is demonstrated in Fig. 2.

It can be seen from the presented dependence that the greatest angle mismatch is observed in the long-wavelength region of the spectrum. Therefore, in this case, the period of the multilayer mirror should be chosen in such a way that at the longest wavelength from the operating range of the monochromator, the angles corresponding to the maximum reflectance for the crystal and the mirror coincide. At the same time, the permissible angle mismatch should not exceed 0.34 degrees due to the design of the monochromator and the recording part. Numerical calculation shows that for the angle corresponding to the first Bragg peak W/B4C of the structure at the wavelength 24.8° to be 68.8 degrees, its period (d) should be equal to 13.36 Å. A similar calculation shows that to replace the RbAp crystal with a W/B4C mirror, its period should be 12.96 Å.

2. Experimental findings

The multilayer mirrors synthesized by magnetron sputtering in a cylindrical vacuum chamber equipped with planar

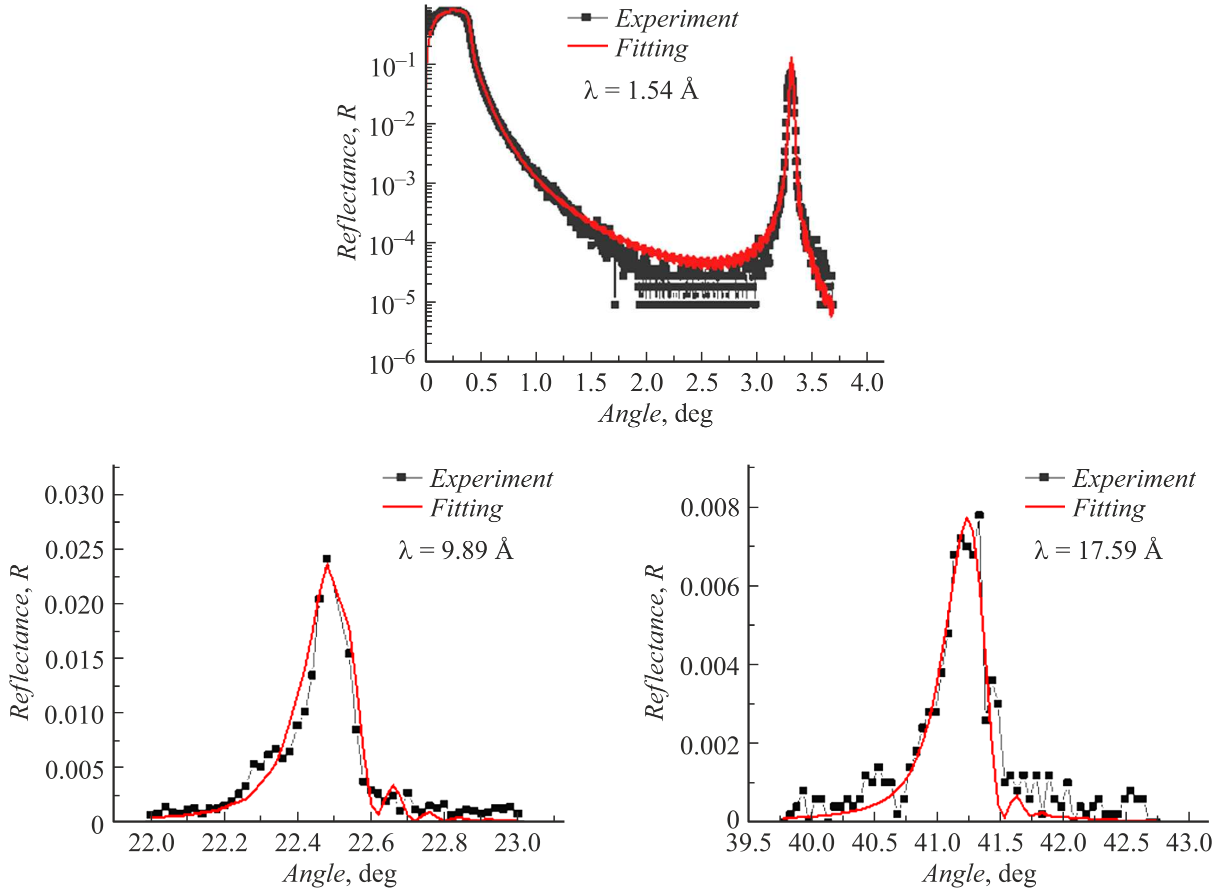

Figure 3. The angular dependences of the reflectance of the multilayer structure W/B4C, designed to replace the KAP crystal, measured at wavelengths 1.54 Å (top), 9.89 Å (bottom left) and 17.59 Å (bottom right). The black curve with dots corresponds to experimental data, the red curve (in online version) matches the fitting.

magnetrons. Stabilized current power supplies developed at IPM RAS were used as magnetron power sources. The working gas was high-purity argon (99.998%), the pressure of residual gases at the time of synthesis of the structure was at the level of 10^{-7} Torr, the pressure of the working gas — at the level of 10^{-3} Torr. The thickness of the layers of materials was changed by changing the speed of passage of the substrate over the targets of the sprayed materials. The sprayed materials was deposited on silicon substrates with a mean square roughness 0.1–0.2 nm. Details about the methods of synthesis and research of multilayer mirrors used in the IPM RAS can be found in [11].

The multilayer mirror designed to replace the KAP crystal was synthesized at the first stage of the experiments. This structure had the following parameters: period $d = 13.36$ Å, tungsten layer thickness $d_W = 5.83$ Å, boron carbide layer thickness $d_{B4C} = 7.53$ Å, roughness of tungsten layers $\sigma_W = 2.5$ Å, roughness of boron carbide layers $\sigma_{B4C} = 4.0$ Å, thickness loss at each period $dz = 0.01\%$ of the nominal value. The structure parameters were determined by fitting the angular dependencies of the reflectance at wavelengths 1.54, 9.89 and 17.59 Å in the Multifitting program developed at the IPM RAS [12]. The above angular dependences, as well as their fitting, are shown in Fig. 3.

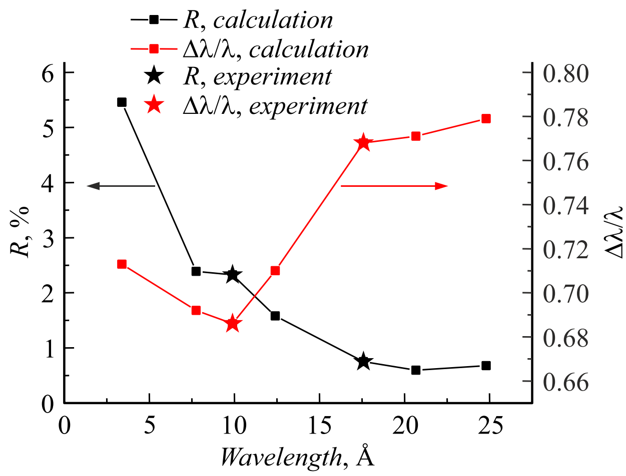

Figure 4. The calculated (dots) and experimental (stars) dependences of the reflectance (black curve) and spectral selectivity (red curve (in the online version)) in the operating wavelength range of a monochromator for a W/B4C structure with a period of $d = 13.36$ Å.

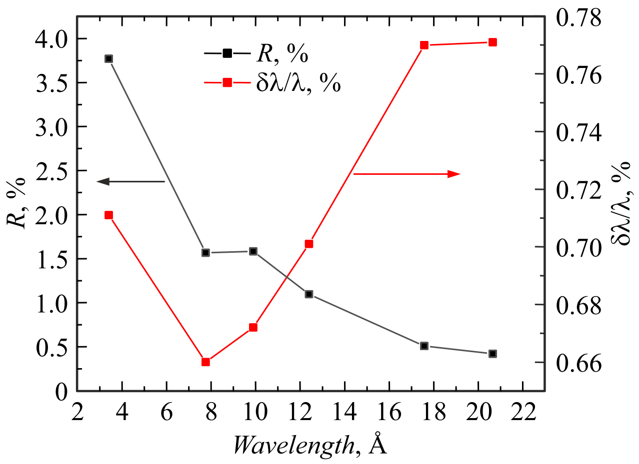

Figure 5. Calculated curves of reflectance (black curve) and spectral selectivity (red curve (in the online version)) in the operating wavelength range of the monochromator for a W/B4C structure with a period of $d = 12.96$ Å.

According to the results of fitting, Fig. 4 shows the calculated (squares) and experimentally measured (stars) dependences of the reflectance and spectral selectivity of a W/B4C mirror with a period of $d = 13.36$ Å of the wavelength. It can be seen from the presented measurement results that the reflectance is at the level of 1−5%, and the spectral selectivity is at the level of 1−2% in most of the range, with the exception of the neighborhood of 45°, where the reflectance of the p-polarized radiation component is close to zero. It is also seen that the mismatch of the Bragg angles in this case lies within the permissible values, which indicates the possibility of using this mirror as a replacement for the KAP crystal in the optical scheme of a two-crystal monochromator.

A multilayer mirror with a period of $d = 12.96$ Å was synthesized in the optical scheme of the monochromator to replace the RbAp crystal. The curves of the reflectance and spectral selectivity (Fig. 5) vs. the wavelength are constructed based on the results of studies of reflectances at the above wavelengths and fitting. The comparison of Figs. 4 and 5 shows that a multilayer mirror with $d = 13.36$ Å looks more preferable from the point of view of the reflectance.

Conclusion

Multilayer mirrors based on a pair of materials W/B_4C were synthesized and studied in this study to use them instead of the first crystal in a two-crystal monochromator circuit at the „KOSMOS“ station. Optimal parameters of mirrors for replacing KAP and RbAp crystals were determined. It was found that the value of the angle at which the maximum reflectance is observed at a given wavelength shows a good match with the corresponding value for crystals for synthesized structures. The misalignment of the angles does not exceed the maximum allowable value, which indicates the prospects of using these structures in the monochromator scheme. The „multilayer mirror system with $d = 13.36$ Å — the KAP“ crystal looks more preferable for solving the task from the point of view of the reflectance.

Funding

This paper was supported by the Russian Science Foundation grant 21-72-30029.

Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] P.S. Zavertkin, D.V. Ivlyushkin, M.R. Mashkovtsev, A.D. Nikolenko, S.A. Sutormina, N.I. Chkhalo. Optoelectronics, Instrumentation and Data Processing, **55** (2), 107 (2019). DOI: 10.3103/S8756699019020018

[2] K. Yamashita, M. Watanabe, O. Matsudo, J. Yamazaki, I. Hatsukade, T. Ishigami, S. Takahama, K. Tamura, M. Ohtani. Rev. Scientific Instruments, **63**, 1217 (1992). DOI: 10.1063/1.1143087

[3] G. Laan, J. Goedkoop, J. Fuggle, M. Bruijn, J. Verhoeven, M.J.V. Wiel, A. MacDowell, J. West, I. Munro. Nucl. Instrum. Methods Phys. Res. Section A-accelerators Spectrometers Detectors and Associated Equipment, **255** (3), 592 (1987). DOI: 10.1016/0168-9002(87)91229-0

[4] S. Bajt, D. G. Stearns. Appl. Opt., **44** (36), 7735 (2005). DOI: 10.1364/AO.44.007735

[5] P.C. Pradhan, A. Majhi, M. Nayak. J. Appl. Phys., **123**, 095302 (2018). DOI: 10.1063/1.5018266

[6] S.S. Andreev, M.S. Bibishkin, N.I. Chkhalo, E.B. Kluenkov, K.A. Prokhorov, N.N. Salashchenko, M.V. Zorina, F. Schafers, L.A. Shmaenok. J. Synchrotron Radiation, **10** (5), 358 (2003). DOI: 10.1107/S0909049503015255

[7] M.S. Bibishkin, N.I. Chkhalo, A.A. Fraerman, A.E. Pestov, K.A. Prokhorov, N.N. Salashchenko, Yu.A. Vainer. Nucl. Instrum. Methods in Phys. Res. A, **543**, 333 (2005). DOI: 10.1016/j.nima.2005.01.251

[8] Ch. Borel, Ch. Morawe, E. Ziegler, T. Bigault, J.-Y. Massonnat, J.-Ch. Peffen, E. Debourg. Laser-Generated, Synchrotron, and Other Laboratory X-Ray and EUV Sources, Optics, and Applications II, **5918**, 591801 (2005). DOI: 10.1117/12.613873

[9] P.N. Rao, S.K. Rai, M. Nayak, G.S. Lodha. Appl. Opt., **52** (25), 6126 (2013). DOI: 10.1364/AO.52.006126

[10] P.S. Singam, M. Nayak, R. Gupta, P.C. Pradhan, A. Majhi, Sh. Narendranath, P. Sreekumar. J. Astron. Telesc. Instrum. Syst., **4** (4), 044003 (2018). DOI: 10.1117/1.JATIS.4.4.044003

[11] A.D. Akhsakhalyan, E.B. Kluenkov, A.Ya. Lopatin, V.I. Luchin, A.N. Nechay, A.E. Pestov, V.N. Polkovnikov, N.N. Salashchenko, M.V. Svechnikov, M.N. Toropov, N.N. Tsybin, N.I. Chkhalo, A.V. Shcherbakov. J. Surf. Investigation: X-ray, Synchrotron and Neutron Techniques, **11**, 1 (2017). DOI: 10.1134/S1027451017010049

[12] M. Svechnikov. J. Appl. Crystallogr., **53** (1), 244 (2020). DOI: 10.1107/S160057671901584X

Translated by A.Akhtyamov